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QUARTERLY REPORT NO. 22

PROJECT NO. A-212

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HIGH TEMPERATURE CERAMIC STRUCTURES

By

N. E. POULOS, S. R. ELKINS, AND J. D. WALTON

CONTRACT NO. NOrd-15701
DEPARTMENT OF THE NAVY
BUREAU OF NAVAL WEAPONS

1 NOVEMBER 1961 TO 31 JANUARY 1962

Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia

QUARTERLY REPORT NO. 22

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I. SUMMARY

The effort during this quarter was directed towards two phases of work, aluminum chloride impregnating studies and radome fabricating studies. The larger effort was expended in the fabricating of radomes from slip-cast fused silica.

The aluminum chloride impregnating studies were continued from the previous quarter to determine the impregnating method that would increase the strength and decrease the porosity of slip-cast fused silica. Four different impregnating methods were evaluated and found effective in decreasing porosity of slip-cast fused silica in the following order, the most effective listed first: (1) simple exposure, (2) boiling, (3) vacuum-pressure, and (4) pressure; and the order according to effectiveness in increasing transverse strength was (1) vacuum-pressure, (2) pressure, (3) simple exposure, and (4) boiling.

The effects of surface skin (fine particles of fused silica developed during initial casting) removal were studied in conjunction with the aluminum chloride studies. In general, the removal of the surface skin decreased the strength of the slip-cast fused silica impregnated by the four impregnating methods studied. The bulk diffusion rate and the tendency to develop closed pores was decreased.

A procedure was developed for pressure casting radomes from fused silica slips. This procedure provided a casting time of 80 minutes which was an approximate reduction of 9 hours in casting time using conventional slip-casting procedures to form a 3/8-inch thick x 15-inch diameter x 32-inch high radome.

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A furnace to facilitate firing large radome shapes was designed and constructed. It is capable of firing slip-cast fused silica radomes of 15-inches in diameter by 32-inches in height to a temperature to 2600° F. Radomes can be loaded or unloaded in this furnace in approximately 2 minutes with a drop in furnace temperature during this period of only 150° F.

II. PURPOSE

The purpose of Contract No. NOrd-15701, as amended, is to develop missile structural components for use in critical structural regions capable of operation at high stagnation temperatures under the combined parameters of thermal shock, high pressures and oxidizing atmospheres.

III. EXPERIMENTAL WORK

The effort during this quarter was directed towards two phases of work, aluminum chloride impregnating studies and radome fabricating studies. The larger effort was expended in the fabrication of radomes from slip-cast fused silica.

A. Impregnation Studies

The aluminum chloride impregnating studies, described in Summary Report No. 5 of the previous contract year, was continued. Emphasis was placed on the evaluation of the four impregnating methods described below. These methods included simple exposure, boiling, vacuum-pressure and pressure. The boiling method had preliminarily appeared to be the most promising and particular emphasis was placed on the evaluation of this method.

Included with these studies was the investigation of the effects of the thin surface skin, developed during initial casting from fine particles of fused silica, on the impregnation of the silica. This thin skin was removed from the fired test bars for the below described investigations by wetting the bars thoroughly with water and then rolling the bars in a gallon jar with a water slurry of -200 mesh alundum grain for approximately 2 hours.

All test bars used in this study were 3/4-inch diameter x 5-5/8-inch long slip-cast fused silica. These bars were dried and given a preliminary firing at 1800° F for 2 hours in an electric kiln. The bars were shielded from the heating elements during firing with a 24 gauge Inconel sheet. The bars were then divided into two groups, skinned and unskinned, these two groups were divided into two sub-groups, impregnated and standard.

These group divisions were necessary to obtain a correlation of the effect of the impregnant on the physical properties of slip-cast fused silica and the effect of the previously mentioned thin skin on the impregnation of silica. The standard bar group was set-up for both the impregnated and skinned bars to obtain a datum resulting from the additional firing treatment given each group.

The two major groups of bars, skinned and unskinned, were divided into 5 groups of 20 bars each and were designated as A, B, C, D, and standard for the unskinned bars; and A-1, B-1, C-1, D-1, and standard-1 for the skinned bars. These designations, with the exception of the standard and standard-1, were the impregnating methods used and are described in Table I. The only treatment for the standard and standard-1 bars was that they were fired under the same conditions and temperatures as A through D and A-1 through D-1.

TABLE I
IMPREGNATING METHODS STUDIED

Group	Impregnating Methods
	<u>Simple Exposure</u>
A and A-1	The bars were placed vertically in a 50 per cent aluminum chloride ($\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$) solution for 24 hours and then immediately placed in a 1 N ammonium hydroxide solution for 24 hours to precipitate the alumina in the voids of the bars.

(Continued)

TABLE I (Continued)
IMPREGNATING METHODS STUDIED

<u>Group</u>	<u>Impregnating Methods</u>
	<u>Boiling</u>
B and B-1	The bars were boiled in a 50 per cent aluminum chloride solution for 2 hours, cooled to room temperature, and then boiled in a 1 N ammonium hydroxide solution for 2 hours and cooled to room temperature.
	<u>Vacuum - Pressure</u>
C and C-1	The lateral surface of each bar was sealed with tape and the ends were left exposed. Then for 16 hours, one end of each bar was exposed to a vacuum 28 inches of Hg while the other end and the remainder of the bar was submerged in a 50 per cent aluminum chloride solution under a pressure of 40 psig. The treatment was then repeated using a 1 N ammonium hydroxide solution.
	<u>Pressure</u>
D and D-1	The bars were treated in the same manner as the bars of groups C and C-1, except that: one end of each bar was exposed to the atmosphere, while the remainder of the bar was submerged in the impregnating fluid under a pressure of 55 psig.

All of the bars were prepared for firing, fired and cooled to room temperature as described below:

- (1) After impregnation, all bars were dried at 130° F for 8 hours dried at 230° F for 16 hours.

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- (2) The bars were then radomely grouped, using tables of random numbers, into 5 groups of 40 bars each.
- (3) The bars were vertically fired at 2200° F for 4 hours in a bottom loading electric glo-bar kiln shielded with 24 gauge Inconel sheet.
- (4) After firing the bars were placed in a desiccator and stored until they were tested.

Porosity, bulk density, and theoretical density determinations were made on each bar using an air displacement apparatus*. In addition, measurements were made on each bar to determine its weight per cent pickup of the impregnant and its transverse strength.

Table II shows the results of the air displacement measurements made on the 1800° F fused silica bars both before and after their surface skins had been removed.

TABLE II
EFFECT OF SURFACE SKIN ON PHYSICAL PROPERTIES
OF SLIP-CAST FUSED SILICA

No. of Bars	% Pores	Before Removing Surface Skins		% Pores	After Removing Surface Skins	
		Bulk Density (GM/CC)	Theoretical Density (GM/CC)		Bulk Density (GM/CC)	Theoretical Density (GM/CC)
88	17.65	1.852	2.250	17.73	1.852	2.250

Note: The above slip-cast fused silica test bars were 3/4-inch in diameter and fired at 1800° F for 2 hours

* Progress Report No. 3, Project No. B-153, Contract No. AT-(40-1)-2483
U. S. Atomic Energy Commission, J. D. Fleming and J. W. Johnson.

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Although the difference in the porosities of skinned and unskinned bars was small, the difference was found to be significant at greater than the 99.5 per cent confidence level by the "Student t" test. This would indicate that the surface skin of the fused silica bars was denser than the interior of the bars.

The results of the air displacement and weight per cent pick-up measurements for the impregnating study are shown in Tables III and IV.

TABLE III

PHYSICAL PROPERTIES OF SLIP-CAST FUSED SILICA
BEFORE IMPREGNATING WITH ALUMINUM CHLORIDE

<u>Group</u>	<u>No. of Bars</u>	<u>Porosity</u> (%)	<u>Bulk Density</u> (GM/CC)	<u>Theoretical Density</u> (GM/CC)
A	19	17.67	1.852	2.249
B	19	17.66	1.855	2.253
C	16	17.57	1.853	2.248
D	17	17.59	1.852	2.248
Std	20	17.59	1.853	2.248
A-1	16	17.77	1.852	2.253
B-1	18	17.89	1.851	2.254
C-1	19	17.76	1.850	2.250
D-1	18	17.69	1.852	2.249
Std-1	17	17.52	1.853	2.247

Note: Bars of this table were 3/4-inch diameter and were fired at 1800° F for 2 hours.

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TABLE IV

PHYSICAL PROPERTIES OF SLIP-CAST FUSED SILICA
IMPREGNATED WITH ALUMINUM CHLORIDE

<u>Group</u>	<u>No. of Bars</u>	<u>Porosity</u> (%)	<u>Bulk Density</u> (GM/CC)	<u>Theoretical Density</u> (GM/CC)	<u>Weight Alumina Pickup</u> (%)	<u>Transverse Strength</u> (PSI)
A	19	12.52	1.949	2.228	0.052	3060
B	19	11.99	1.950	2.216	0.64	2820
C	16	12.55	1.952	2.230	0.74	4610
D	17	12.49	1.952	2.231	0.63	4340
Std	20	14.57	1.906	2.230	-	3750
A-1	16	12.32	1.954	2.226	0.42	2910
B-1	18	12.38	1.947	2.223	0.69	3110
C-1	19	12.46	1.958	2.236	0.85	4020
D-1	18	12.36	1.957	2.233	0.51	3500
Std-1	17	14.80	1.906	2.237	-	3900

Note: These bars are the bars from Table III but which were
impregnated with aluminum chloride then fired at 2200° F
for 4 hours.

If we let:

Po = per cent porosity before impregnation

Bo = bulk density before impregnation

P = per cent porosity after the 2200° F firing

B = bulk density after the 2200° F firing

W = weight per cent pickup

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Then:

The per cent decrease in open pores after impregnating and firing at 2200° F would be $100 - \frac{P_{Bo}}{P_{oB}} (100 + W)$.

The per cent decrease in open pores due to bulk diffusion alone would be $100 - \frac{B_o}{B} (100 + W) \frac{100}{P_o}$.

The per cent pores filled by the impregnating medium would be $\frac{100 W B_o}{3.96 P_o}$, where 3.96 is the density of Al_2O_3 in gm/cc.

Applying the above calculations to the data obtained for the impregnating study, gives the results shown in Table V.

TABLE V

PER CENT DECREASE IN OPEN PORES UPON IMPREGNATION
WITH ALUMINUM CHLORIDE AND FIRING AT 2200° F FOR 2 HOURS

<u>Group</u>	<u>By Bulk Diffusion</u>	<u>By Filling Pores</u>	<u>By Closing[*] Pores</u>	<u>Total</u>
A	25.4	1.4	5.5	32.3
B	24.1	1.7	9.8	35.0
C	24.9	2.0	4.8	31.7
D	25.8	1.7	4.7	32.2
Std	15.8	-	3.7	19.5
A-1	27.1	1.1	5.8	34.0
B-1	23.9	1.8	8.1	33.8
C-1	26.5	2.2	4.4	33.1
D-1	27.6	1.4	4.5	33.5
Std-1	15.9	-	2.0	17.9

^{*} As determined by difference

Significance tests were not made on the above data. However, indications are that:

1. In all instances the presence of the impregnating medium increased the bulk diffusion rate and promoted the closing of pores during the 2200° F firing.

2. Listed (in order) according to effectiveness in decreasing total porosity, the impregnation methods were

<u>Unskinned Bars</u>	<u>Skinned Bars</u>
(i) The boiling method	The simple exposure method
(ii) The simple exposure method	The boiling method
(iii) The pressure method	The pressure method
(iv) The vacuum-pressure-method	The vacuum-pressure-method

3. In general, the bulk diffusion rate was greater for the bars that had their surface skin removed.

4. The bars that had their lateral surface exposed to the impregnating fluid showed a greater tendency to develop closed pores.

5. The bars that were boiled in the impregnating fluid showed a marked tendency to develop closed pores.

6. Generally, the unskinned bars showed a greater tendency to develop closed pores.

Table IV lists the effect of the alumina impregnant, the impregnating method, and the removal of the previously mentioned thin skin on the strength of slip-cast fused silica.

Significance tests were not made on the strength data. However, indications are that:

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1. The bars that had their lateral surface exposed to the impregnating fluid were weaker than the standard bars. It was noted that these (groups A, B, A-1, B-1) bars showed surface cracks.
2. Generally, the bars impregnated by the pressure method or the vacuum-pressure method were stronger than the standard bars - the vacuum-pressure bars (group C) exhibiting the best strength, M.R. = 4610 psi.

B. Radome Fabrication

The fabrication of the two radome shapes discussed in Summary Report No. 5, Project No. A-212 was continued with emphasis placed on developing pressure casting techniques and the fabrication of a kiln large enough to handle the large shapes.

Radome model B, shown in Figure 1 was used to form the plaster casting mold shown in Figure 2. This plaster mold was formed following the same general procedure used for radome A discussed in Summary Report No. 5, Project No. A-212.

The plaster mold shown in Figure 2 was keltexed by pouring a 0.2 per cent keltex solution into the plaster mold. After a soaking period of approximately three minutes, the excess keltex solution is removed and the deposited film allowed to dry. This procedure provided an excellent method of removing slip-cast radomes from the plaster molds. Radome B was then pressure cast using the setup shown in Figure 3. The reservoir (labeled A in Figure 3) was filled with fused silica slip which was forced by air pressure (≈ 10 psig) through the slip or air intake valve (labeled B) and pressure plate (labeled C) into the plaster mold (labeled D).

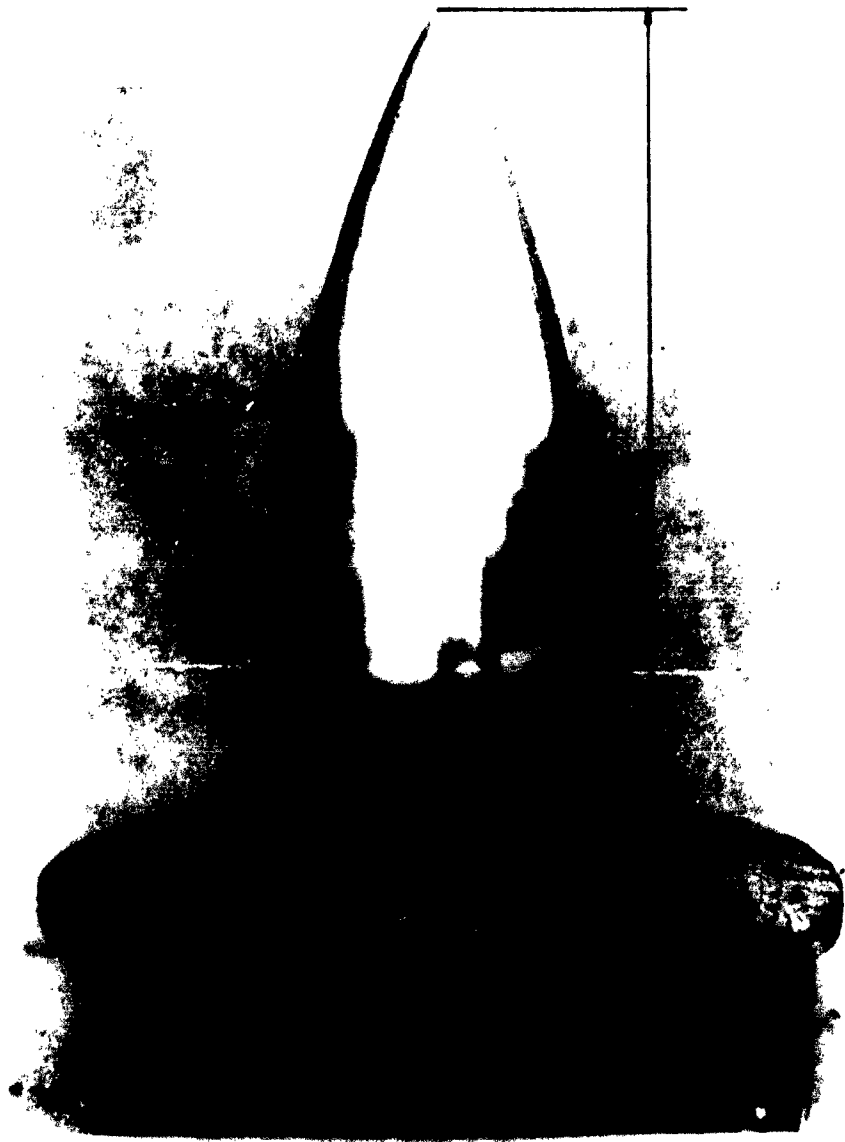


Figure 1. A valve, Model F Range Configuration B Approximately 1
Inches in diameter with an overall height of 1.0 inches.

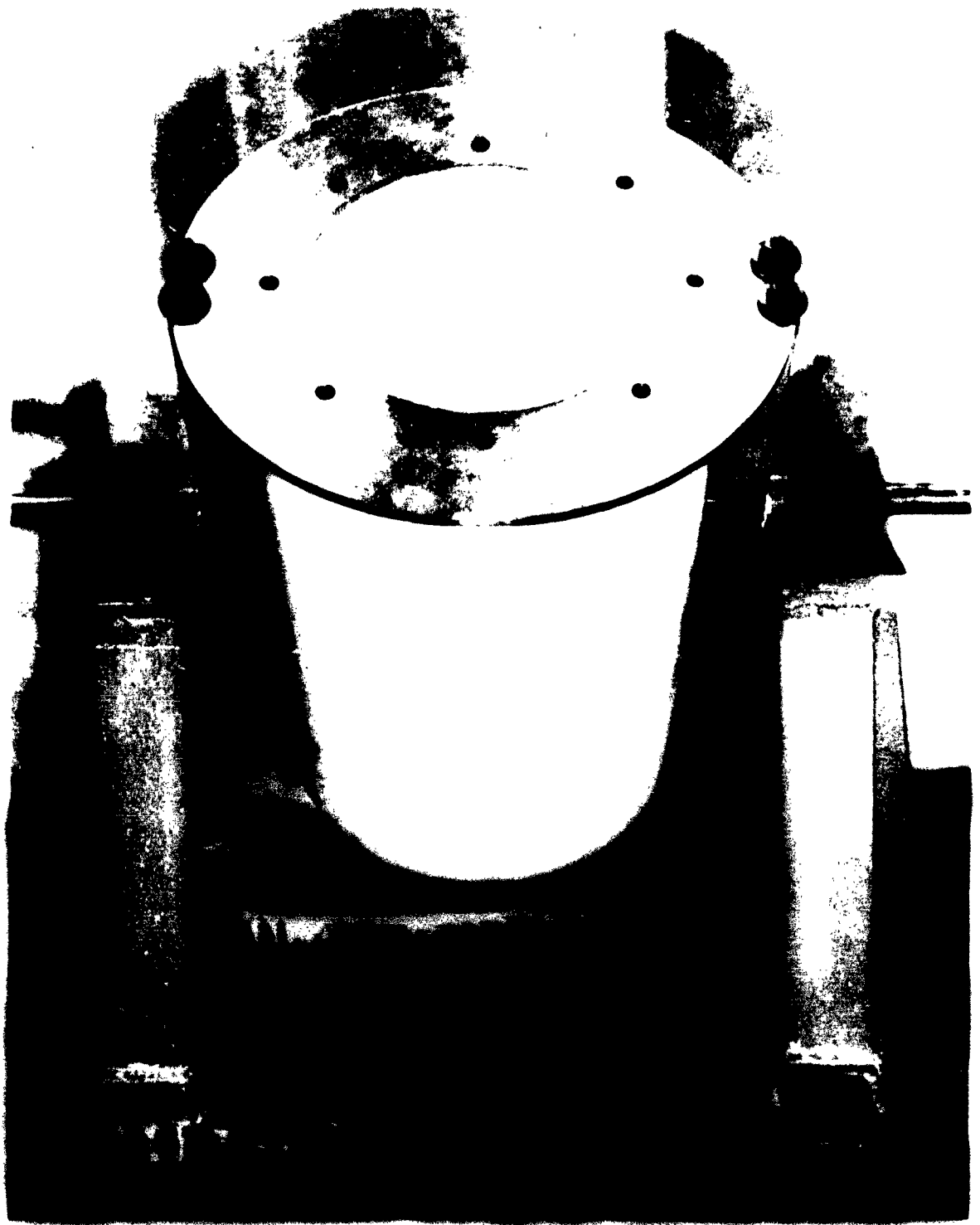


Figure 1. Plate M in CR Line Configuration B Tilted 31° from
Plate Configuration with Mold Support D 113.

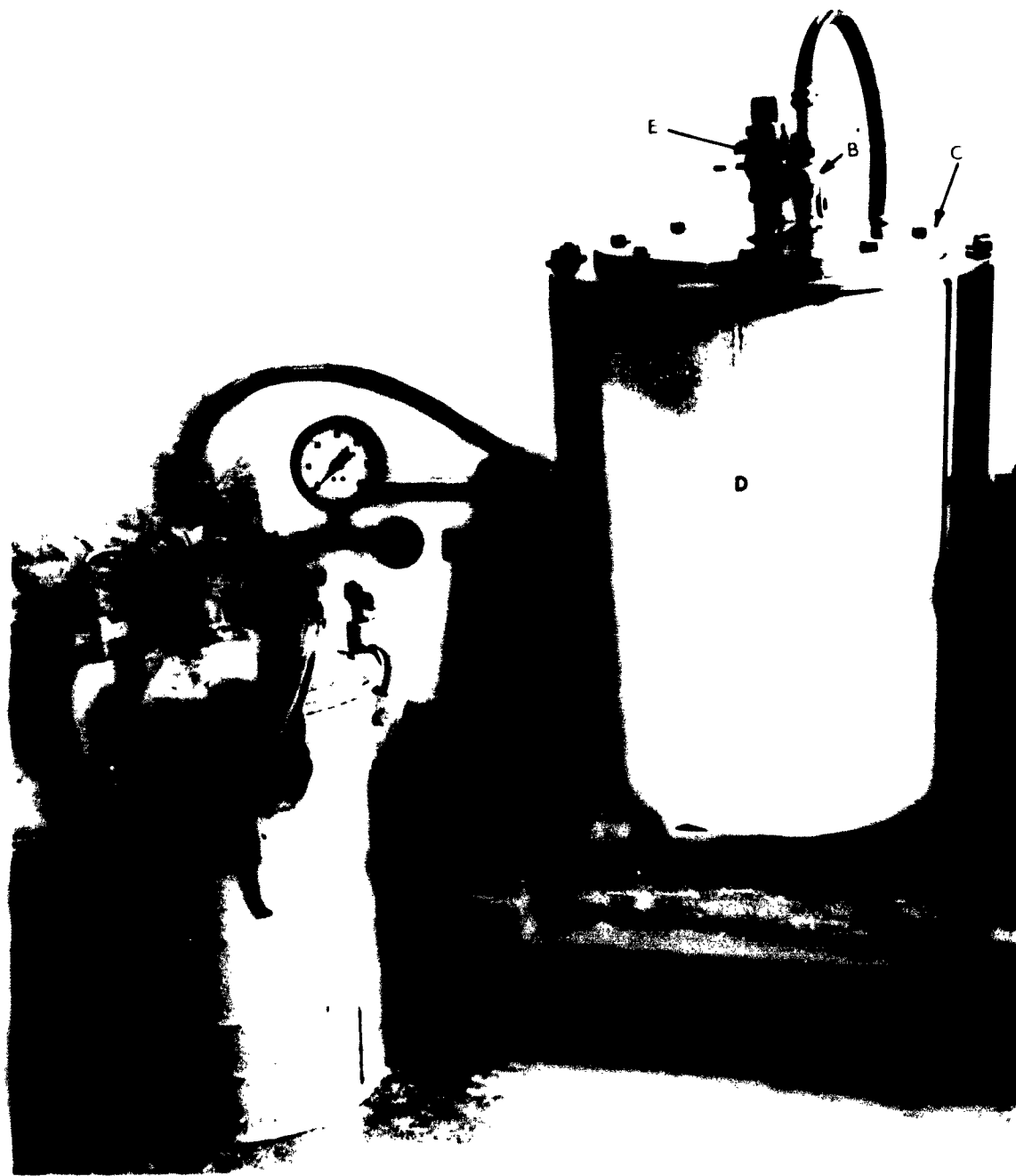


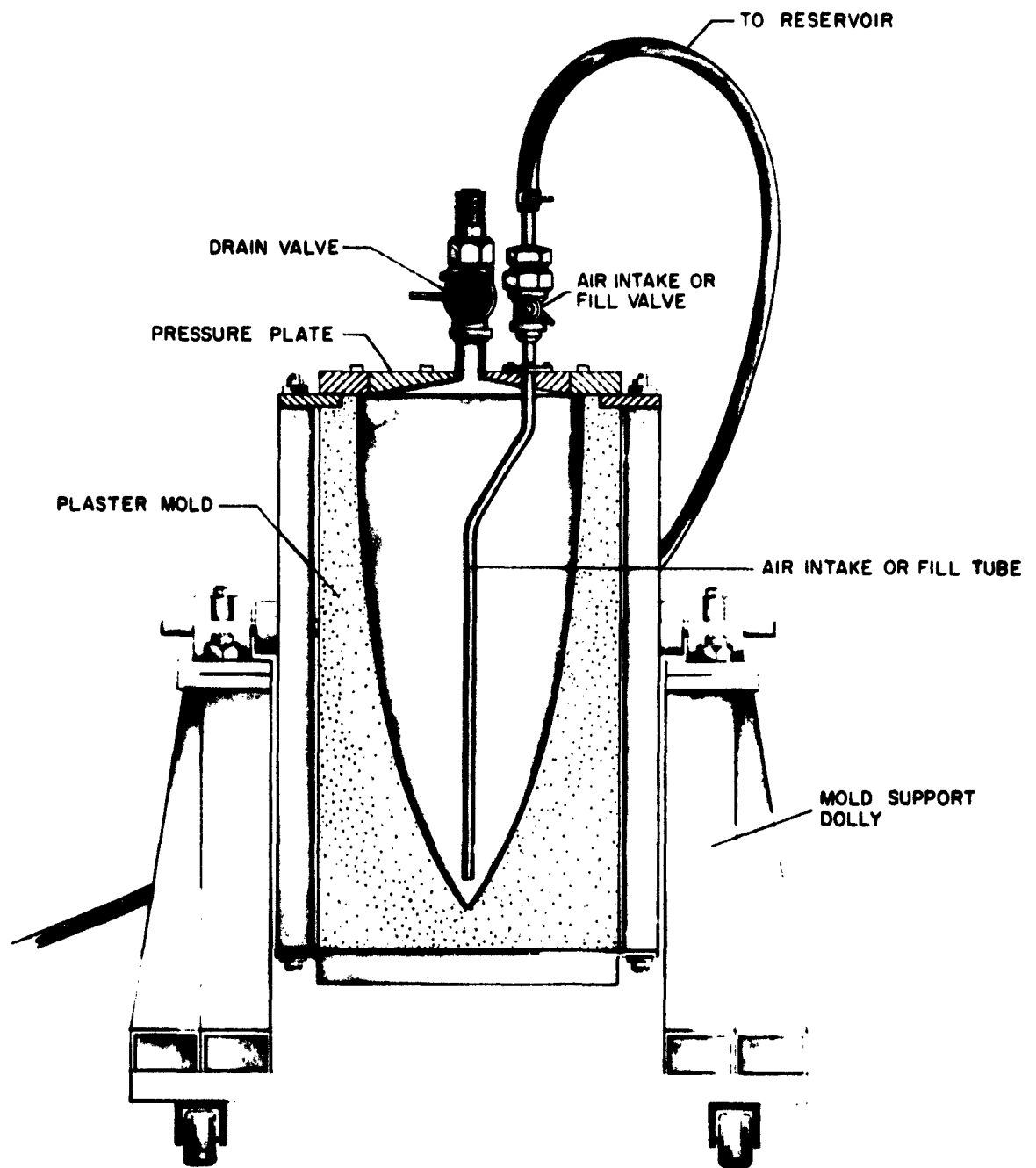
Figure 1. Pressure Gauge Assembly. D. 1. Pressure Gauge. R. 1. Pressure Gauge. C. 1. Pressure Gauge. B. 1. Pressure Gauge. E. 1. Pressure Gauge.

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The slip was allowed to overflow through the drain valve (labeled E) until all the trapped air was removed from the mold. The drain valve was then closed and the entire system was allowed to reach an equilibrium pressure of approximately 20 psig. Upon completion of the casting period (about 1 hour and 20 minutes) the slip fill line was disconnected from the slip or air intake valve and the valve closed. The mold was then inverted and the excess slip removed by opening the air intake and drain valves. The drain rate of the slip was approximately 250 ml/sec. After draining, the pressure plate was removed with the mold still in the inverted position. This was done to prevent any wet slip that may be left on the plate from draining into the cast radome. The radome was extracted from the mold then placed in a dryer in readiness for firing. Figure 4 shows a cutaway view of the plaster mold and pressure plate. Figure 5 shows the slip-cast radome B. The cast wall thickness for radome B for a casting time of about 1 hour and 20 minutes was approximately 3/8-inch.

In order to determine the possibility of reusing the drained fused silica slip, the particle size distribution, per cent solids, and slip density were determined before and after casting. The results are shown in Figures 6 and 7. These results indicate that the slip can be reused from casting to casting.

A large furnace was designed and constructed to accommodate the firing of the large radome shapes. The furnace cavity is in the shape of an octagon with a firing height of 32 inches. Circular ware up to fifteen inches in diameter with an overall height of 32 inches can be fired in the furnace. The furnace is electrically heated using silicon carbide heating elements



p. matrangoes 'ee

Figure 4. Cutaway View of Plaster Mold Used to Pressure Cast Rods B.



PROPERTIES BEFORE CASTING

PER CENT SOLIDS 82.8 %

SLIP DENSITY 1.02 GM/CC

NOTE: AVERAGE OF TWO TRIALS

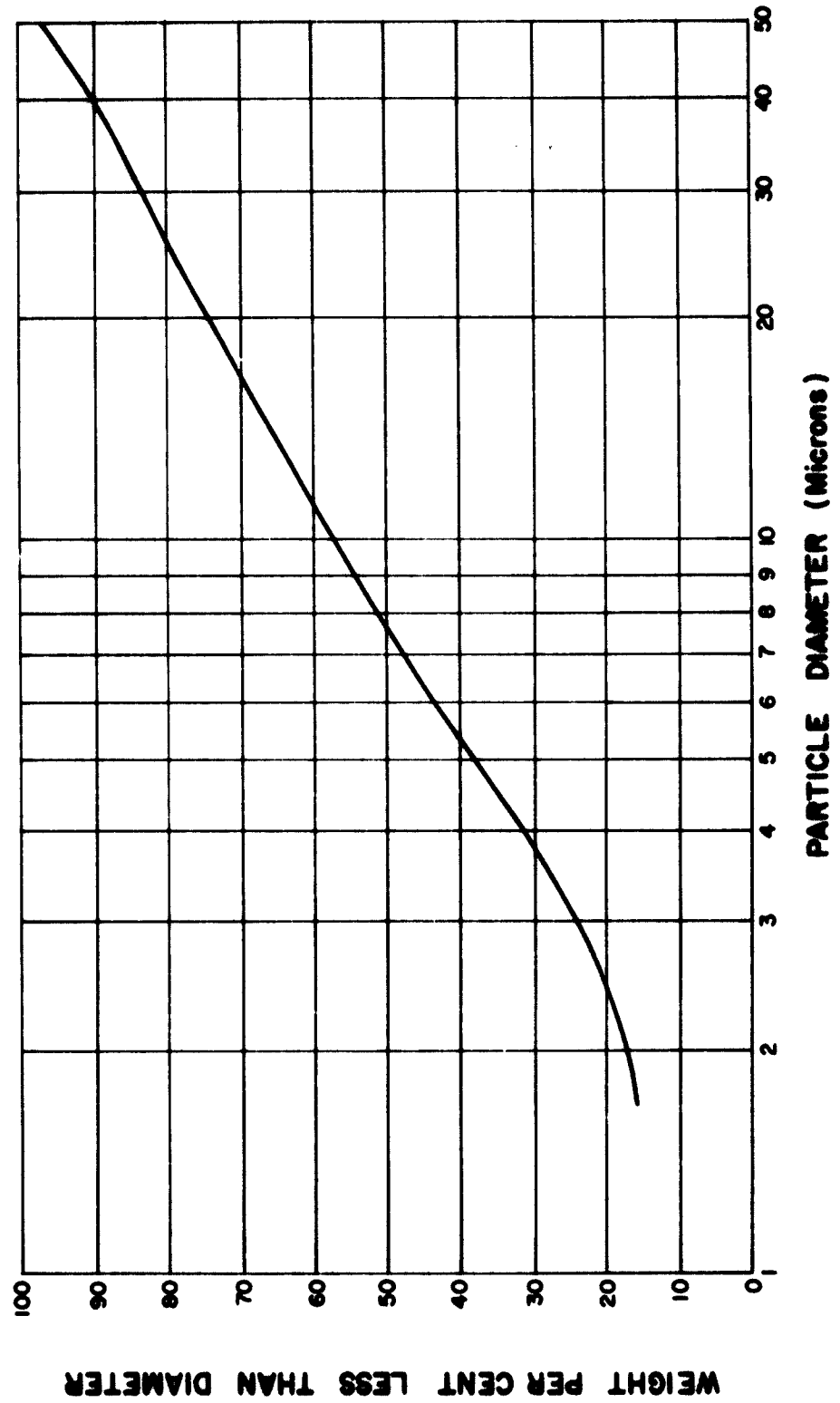


Figure 6. Particle Size Distribution, Per Cent Solids, and Slip Density of Fused Silica Slip Before Pressure Casting.

PROPERTIES AFTER CASTING

PER CENT SOLIDS 82.9 %

SLIP DENSITY 1.83 GM/CC

NOTE: AVERAGE OF TWO TRIALS

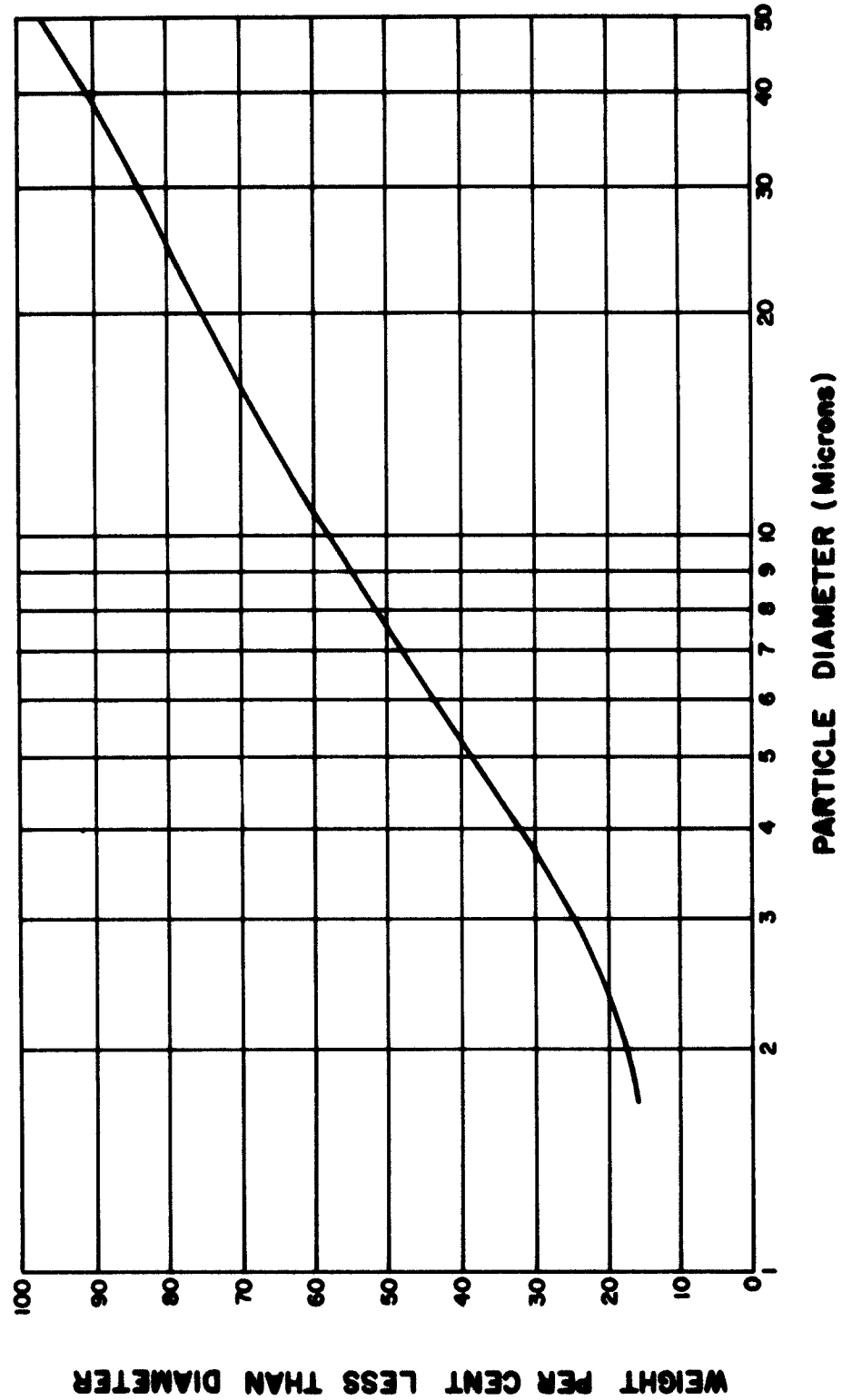


Figure 7. Particle Size Distribution, Per Cent Solids, and Slip Density of Fused Silica Slip After Pressure Casting.

controlled with a saturable reactor. Foamed fused silica (7-1/2 inches thick) is used as the insulation for the top and bottom of the furnace and refractory brick (9 inches thick) are used as the insulation for the sides of the furnace. The furnace is loaded from the bottom utilizing a counter balance floating platform system. This arrangement is used so that the radomes can be loaded or unloaded with ease with a minimum loss of heat. Figure 8 shows the complete furnace with the loading platform in the firing position. Figure 9 shows the fused silica radome on the transfer dolly prior to transferring to platform while Figure 10 shows the dolly in the same position with the Inconel shield in place. Figure 11 shows the dolly on the platform prior to loading in the furnace.

The following procedure was developed for loading and unloading the fused silica radomes in the furnace:

1. Place radome from dryer room onto radome dolly and roll to furnace area on transfer dolly.
2. Place Inconel firing shield around radome.
3. Remove dummy or plug dolly from the furnace by lowering loading platform and rolling the dummy dolly off the loading platform onto transfer dolly.
4. Roll radome dolly into position in front of furnace and transfer radome dolly onto loading platform and center.
5. Raise loading platform until the firing position is reached which is determined by stops.
6. After firing the radome under the desired conditions, reverse the procedure to unload the furnace.

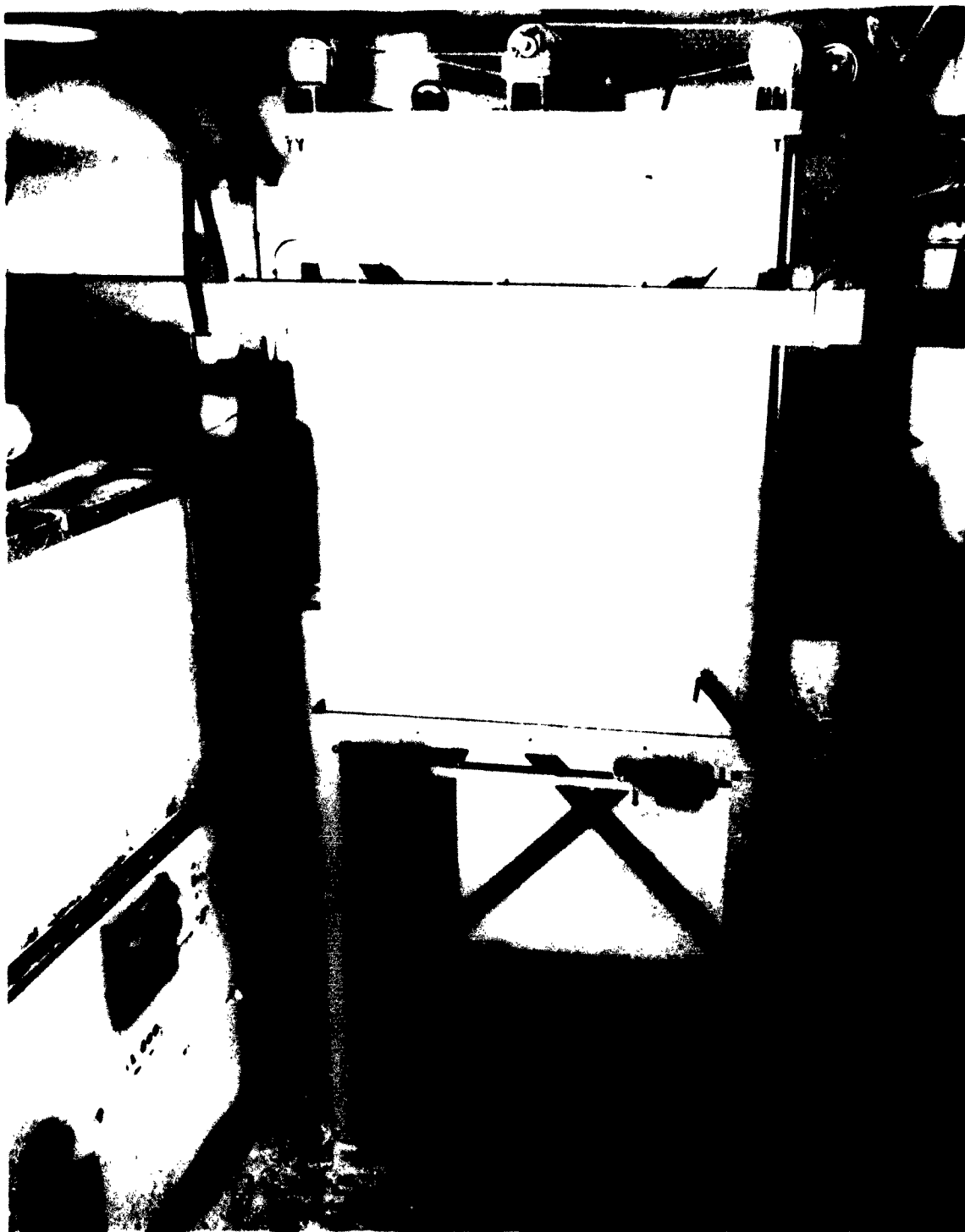


FIGURE 1. (a) View of the Fuel Tank and Fuel System.



Figure 1. Transformer Ref. No. A-212, Transformer D-11, Ref. No. 1, Transformer
D-11, Ref. No. 1, Transformer D-11, Ref. No. 1.



Figure C. Paced Motion Release A with Incident Film Shield in Transfer Dolly Prior to Transferring to Loading Platform.



Figure 1. A view of the R-10-A with the no. 1 film shield centered
on the top of the R-10-A. The R-10-A is in the R-10-A.

By following the above procedure the furnace can be loaded or unloaded in approximately two minutes with a drop in temperature of only about 150° F.

IV. CONCLUSIONS

A. Impregnation Studies

The results of studying skinned vs. unskinned slip-cast fused silica tests bars indicated that there was a small difference in the porosity of the bars (17.65 per cent for unskinned bars and 17.73 per cent for skinned bars). Although the difference in the porosities was small, the difference was found to be significant at greater than the 99.5 per cent confidence level by the "student t" test. The results indicate that the surface skin of the fused silica bars, when slip-cast, is denser than the interior of the bars.

Significance tests were not made on the data obtained for the study of aluminum chloride impregnating methods. However, indications are that:

1. In all instances the presence of the impregnating medium increased the bulk diffusion rate and promoted the closing of pores during the 2200° F firing.
2. Listed (in order) according to effectiveness in decreasing total porosity the impregnating methods were:

Unskinned Bars

Skinned Bars

- | | |
|---------------------------------|----------------------------|
| (i) The boiling method | The simple exposure method |
| (ii) The simple exposure method | The boiling method |
| (iii) The pressure method | The pressure method |
| (iv) The vacuum-pressure method | The vacuum-pressure method |
3. In general, the bulk diffusion rate was greater for the bars that had their surface skin removed.

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4. The bars that had their lateral surface exposed to the impregnating fluid showed a greater tendency to develop closed pores.
5. The bars that were boiled in the impregnating fluid showed a marked tendency to develop closed pores.
6. Generally, the unskinned bars showed a greater tendency to develop closed pores.

The results of the evaluation of strength data indicate that:

1. The bars that had their lateral surface (groups A, A-1, B, B-1) exposed to the impregnating fluid were weaker than the standard (unimpregnated) bars. It was noted that surface cracks developed on the surface of bars impregnated by simple exposure and boiling methods for both the skinned and unskinned bars.
2. Generally, the bars impregnated by the pressure method or the vacuum-pressure method were stronger than the standard bars - vacuum-pressure bars exhibiting the best strength, M.R. = 4610 psi.

The vacuum-pressure or pressure method of impregnation employing unskinned bars proved to provide the best combined effect of decreasing porosity and increasing transverse strength for slip-cast fused silica.

B. Radome Fabrication

The procedure developed for pressure casting radomes from fused silica slip proved very satisfactory. The casting time was reduced from approximately 10 hours (normal casting procedures) to approximately one hour and 20 minutes following pressure casting procedures - applied pressure of 20 psig.

The possibility of reusing the drained fused silica slip was investigated. The results of this investigation indicate that the slip can be reused from casting to casting thus injecting great economy into the system.

The large furnace described in the text of this report proved very satisfactory in firing large radome shapes. The furnace was designed to offer ease in loading or unloading of the radomes with a minimum loss of heat. By following the procedure described, the furnace can be loaded or unloaded in approximately two minutes with a drop in temperature of only about 150° F.

V. PROGRAM FOR THE NEXT INTERVAL

The rate of effort will be reduced by 25 per cent during the next period. This reduction is necessary to compensate for the increased rate expended during this period for the development of procedures for forming and firing the larger, 15-inch diameter - 32-inch high, fused silica radomes.

The greatest effort during this next period will be expended for the below described arc-plasma flame glazing studies.

A. Arc-Plasma Flame Glazing

Investigations will be attempted to establish procedures for glazing small slip cast-fused silica radome shapes. Such procedures will then be expanded for larger shapes.

Several test shapes will be glazed to study the effect of flame glazing on electrical properties and rain-erosion resistance of slip-cast fused silica. These properties will be evaluated by General Dynamics, Pomona Division at no cost to this project.

B. Fused-Silica Pressure Casting

The parameters that govern pressure casting rates of fused silica slip will be studied. These parameters will be established first with forming small test bars. The parameters will then be applied to forming larger shapes such as radomes.

C. Surface Sealing of Fused Silica with Organic Silicates

Preliminary studies will be initiated to determine the feasibility of sealing the surface of slip-cast fused silica by impregnating its surface with aqueous organic silicate solutions. This is a new experimental material

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from Philadelphia Quartz Company. Its solubility in water and the low temperature necessary to destroy its organic complex leaving pure silica as a residue makes this material attractive for sealing slip-cast fused silica.

VI. PERSONNEL

The personnel assigned to the project and the approximate time devoted to the work of the project by each are listed below:

N. E. Poulos	Project Director	1/2 time
J. D. Walton	Special Research Engineer	1/10 time
S. R. Elkins	Assistant Project Director	Full time
C. A. Murphy	Research Assistant	1/2 time
G. L. Maynard	Technician	3/4 time
R. D. Archer	Technician	Full time
W. M. Linstrom	Technician	1/2 time
P. Boland	Graduate Research Assistant	1/5 time
P. S. Matrangos	Technical Illustrator	1/5 time

Respectfully submitted:

N. E. Poulos

N. E. Poulos
Project Director

Approved:

J. D. Walton

J. D. Walton, Head
Ceramics Branch

Frederick Bellinger

Frederick Bellinger, Chief
Material Sciences Division

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